| EFFECTIVE DATE<br>May 27, 1997 | LANL-CST-DP-112, R0<br>Page 1 of 8 |
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# SORPTION OF RADIONUCLIDES ONTO COLLOIDS

***LOS ALAMOS***
***QUALITY PROGRAM***

| APPROVAL FOR RELEASE | |
|---|---|
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**Los Alamos**
**Yucca Mountain Site**
**Characterization Project**

# HISTORY OF REVISION

| REVISION NO. | EFFECTIVE DATE | PAGES REVISED | REASON FOR CHANGE |
|---|---|---|---|
| R0 | 05/27/97 | N/A | Initial procedure. |

# SORPTION OF RADIONUCLIDES ONTO COLLOIDS

## 1.0 PURPOSE

The purpose of this procedure is to delineate laboratory methods for determining sorption parameters of radionuclides onto colloidal particles in a natural or synthetic water.

## 2.0 SCOPE

This procedure defines the process to be used for studies of sorption of radionuclides onto colloidal particles for tasks performed by the Los Alamos National Laboratory (Los Alamos) Yucca Mountain Project (YMP).

## 3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting Scientific Investigations
LANL-YMP-QP-08.1, Identification and Control of Samples
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards
LANL-YMP-QP-17.6, Records Management
LANL-INC-DP-35, pH Measurements
LANL-CST-DP-63, Preparation of NTS Core Samples for Crushed Rock Experiments
LANL-CST-DP-101 Colloid Sampling for YMP Studies
CST Division Environmental Safety and Health Operational Statement
LANL-CST-DP-107, Use of ICP Atomic Emission Spectrometry to Determine Constituent Concentrations in Solution
LANL-CST-DP-79, Liquid Scintillation Counting of Samples

## 4.0 DEFINITIONS

### 4.1 Colloidal Solution

A system intermediate between a true solution and a suspension. A dispersion where the particle size is less than 100 nm. Colloids have little or no tendency to dialyze, and small or no freezing-point depression.

### 4.2 Tracer Solution

Tracer solutions are comprised of either synthetic water or natural water along with a specified concentration of the species whose sorption behavior is to be studied (i.e. plutonium, neptunium, or uranium).

### 4.3 Kd Value

A distribution coefficient of a radionuclide species between solid phase (i.e. colloidal particles) and liquid phase at equilibrium.

$$Kd = \left[\frac{(C_o - C_I)}{C_I}\right]\left[\frac{S}{A}\right]$$

$C_o$: Concentration or radioactivity of the initial tracer in the solution before sorption or desorption.

$C_I$: Concentration or radioactivity of the tracer in the solution after sorption or desorption.

S: Milliliters of solution in contact with colloids.

A: Amount of colloids used in (g).

The Kd values are adjusted when working with colloidal tracers (i.e. when working with Pu IV colloids) because of the effects of centrifuging and sorption effects on the filters.

## 5.0 Responsibilities

The following personnel are responsible for the activities identified in Section 6.0 of this procedure:

- Principle Investigator (PI)
- Users of this Detailed Procedure (DP)

## 6.0 Procedure

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or the current work practice is documented in accordance with QP-03.5, Section 6.1.6.

- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.

- When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

## 6.1 Principle

This procedure can be used to study sorption of a species onto colloids.

## 6.2 Equipment and Hardware / Software

- Calibrated balances
- Scanning Electron Microscope
- Particle Size Analyzer
- X-Ray Diffractometer
- UV-Vis Analyzer
- Analytical pipettes
- ICP / AES
- Centrifuge capable of 15,000 RCF (Relative Centrifugal Force)
- 5 cc Syringes
- 0.2µm and 0.45µm filters
- Liquid Scintillation Counter
- Sonifier / Cell Disruptor

### 6.2.1 Equipment Malfunctions

There are no critical equipment malfunctions that are likely to occur that would interrupt this experiment.

### 6.2.2 Safety Considerations

The samples generated by this procedure may be radioactive; therefore, they should be handled in accordance with CST Division Environmental Safety and Health Operational Statement. The centrifuges must be operated in accordance with the manufacturer's instructions.

### 6.2.3 Special Handling

Control microbial growth in the colloidal solution by storing the colloidal solution in a refrigerator if the experiment cannot be carried out immediately after purifying the colloids.

Ensure that samples are stored in a secondary container. All vials should be placed in a tray with lid for transport to the liquid scintillation analyzers or to the centrifuges. Dispose of according to pertinent laboratory regulations for disposal of radionuclide-containing organic scintillators.

## 6.3 Preparatory Verification

Notebook Entries

The recording of data in the laboratory notebooks or binders can be performed by following the example spreadsheet templates on Attachment 2 and 3.

6.3.1 Hold Points

None

6.3.2 Calibration

Balances used for weighing are required to be calibrated according to QP-12.3. When data are collected from a balance the unique identifier number of that balance is recorded in the user's laboratory notebook along with the data collected. pH meters are calibrated before measurements are taken. To ensure accuracy of sample pH measurements, buffers used for calibration should be measured prior to and after taking sample pH measurements. The unique identifier number of each pH meter used is recorded in the laboratory notebook or binder. (See Attachment 2). The ICP is calibrated according to DP-107.

6.3.3 Environmental Conditions

The first part of the experiment is performed under normal environmental conditions. A second part of the experiment is performed in a glove box under an argon atmosphere.

## 6.4 Control of Samples

Samples are processed in accordance with the following procedures:

- DP-63, Preparation of NTS Core Samples for Crushed Rock Experiments
- QP-08.1, Identification and Control of Samples

## 6.5 Implementing Procedure

6.5.1 Prepare tracer solutions to be used for the experiments and document the method used in the laboratory notebook.

6.5.2 Determine the initial concentration or radioactivity of the tracer solution by radiochemical analysis and measure the pH of the tracer solution.

6.5.3 Prepare the colloidal solution as follows:

6.5.3.1 Weigh a set amount of water into a clean bottle.

6.5.3.2 Add a set amount of the colloidal material to the bottle.

6.5.3.3 Disperse particles for desired time using sonifier/cell disruptor depending on the characteristics of the colloidal material.

6.5.3.4 Allow the suspension to stand for at least 3 hours.

6.5.3.5 Determine the mass of the colloidal particles in the solution by drying in a temperature controlled oven or evaporation, record in mg / ml in lab notebook.

6.5.4 Pipette set amounts of the colloidal solution to previously weighed centrifuge tubes and record the weights of the solution and tube.

6.5.5 Add set amounts of the tracer to the centrifuge tubes from step 6.5.4 and record the total weights.

6.5.6 Make control samples by adding the same amount of natural or synthetic water and the tracer as the samples being tested. The controls will go through all the same steps as your sample.

6.5.7 Shake the suspensions for desired time using an orbital shaker at a setting of 150 rpm.

6.5.8 Centrifuge the samples at 15,000 rpm for at least 2 hours.

6.5.9 Carefully pipette supernates into vials. remove residual solutions carefully with a transfer pipette and dispose of properly. Do not remove the colloidal particles when removing residual solutions.

6.5.10 Filter the supernates through double filters (0.45µm and 0.2µm) using a syringe.

6.5.11 Count the filtered solutions from 6.5.10 on the LSC following DP-79.

6.5.12 Weigh the tubes and the wet colloidal particles. Take off lids and allow the wet colloidal particles to dry for at least four days.

6.5.13 Weigh the tubes and the air dried colloidal particles and record weights in lab notebook. Store the air dried colloidal particles for further analysis (i.e. x-ray diffraction or scanning electron microscopy).

6.5.14 If desorption is going to be performed do not air dry the wet colloidal particles. Follow the desorption procedure DP-86.

6.5.15 Test the effects of filtration on the radioactivity of the tracer by filtering the initial tracer solution and then counting on the LSC. Compare the values with a sample of the tracer that is not filtered.

6.5.16 Test the effects of centrifuging on the radioactivity of the initial tracer by comparing the radioactivity of the tracer before and after centrifuging.

6.5.17 Throughout the experiment check the tracer solutions frequently to make sure that there are no formations of precipitates.

### 6.6 Data Acquisition and Reduction

Calculate the Kd according to the definition given in section 4.0.

Calculate the amount of tracer sorbed onto the colloids in pCi/g or pCi/$cm^2$.

### 6.7 Potential Sources of Error and Uncertainty

Sources of error and uncertainty include:

a) Incomplete separation of solid and liquid samples.

b) Leakage from the container during shaking or handling

c) Improper recording or transfer of data

The responsible PI or his/her designee will determine whether to use the collected data. If a decision to use the data is made, the justification for this decision must be entered in the investigator's logbook.

## 7.0 RECORDS

Records generated as a result of this DP are entries in the laboratory notebooks and electronic media on which data is stored. These records are kept in accordance with QP-03.5.

All records should be submitted to the records processing center in accordance with QP-17.6.

## 8.0 ACCEPTANCE CRITERIA

Proper completion and submittal of the records described in Section 7.0 constitute the acceptance criteria for this procedure.

## 9.0 TRAINING

9.1 Prior to conducting work described in Section 6.0, the user requires on the job training and documentation pursuant to QP-02.7.

9.2 Training in handling of samples is covered by QP-08.1 and is read only.

## 10.0 ATTACHMENTS

Attachment 1: Spreadsheet
Attachment 2: Data Sheet

# SPREADSHEET

| Sample # | Sample I.D. | WATER I.D. | Sorption Time | Mass of Tube +Cap (g) | Mass of Tube + Cap + Colloidal Soln. (g) | Mass of Colloidal soln. (g) | Mass of Colloidal Solid in Soln. (mg) | Mass of Tube + Cap +Colloidal Soln. + Tracer Soln. (g) | Mass of Tracer (g) | Total Counts Radio-nuclide added to each tube (CPM) | Mass of Counting Vial (g) | Mass of Vial + Extracts (g) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| N | O | P | Q | R | S | T | U | V | W | X | | |
| Mass of Extracts (g) | Total LSC Counts (CPM ) | Counts per unit extracts (CPM/g ) | Radio-nuclide Left in Soln. After sorption (CPM) | Radio-nuclide Adsorbed by Colloid (CPM/mg) | Radio-nuclide Adsorbed (pCi /g Colloid) | Distribution Coefficients of Radionuclide on Colloid (Kd) | % Radio-nuclide adsorbed | Scintillation Cocktail Used | Tracer Used for Sorption Exp. | pH of Tracer Solution | | |

# DATA SHEET

| YUCCA MOUNTAIN SORPTION DATA INFORMATION SHEET, Fe Colloids | |
|---|---|
| Experiment Number | |
| ID of Water Used in Experiment | |
| Notebook Reference | |
| Tracer Solution ID# | |
| Tracer Solution Information | |
| Notebook with Information on Tracer Solution | |
| YMP Balance ID# Used Before Addition of Tracer | |
| YMP Balance Calibration Due Date | |
| YMP Balance ID# Used after Addition of Tracer | |
| YMP Balance Calibration Due Date | |
| Mineral Code IDs | |
| Sieving Information | |
| Notebook Reference for Information on Solid Samples | |
| **pH Measurement** | |
| pH Meter Used, YMP ID# | |
| Detailed Procedure Used | |
| DP Revision Number | |
| pH Buffer 4, Manufacturer/Lot #/Expiration Date | |
| pH Buffer 4 (Prior to Making Measurements) | |
| pH Buffer 4 (After Making Measurements) | |
| pH Buffer 7, Manufacturer/Lot #/Expiration Date | |
| pH Buffer 7 (Prior to Making Measurements) | |
| pH Buffer 7 (After Making Measurements) | |
| pH Buffer 10, Manufacturer/Lot #/Expiration Date | |
| pH buffer 10 (Prior to Making Measurements) | |
| pH Buffer 10 (After Making Measurements) | |
| Tracer Solution pH Before Adding to OR Tubes | |
| **Verification of Liquid Scintillation Counter** | |
| YMP Counter ID# | |
| ID# of Low Standard Counted | |
| Expected CPM of Standard Counted | |
| ID# of High Standard Counted | |
| Expected CPM of Standard Counted | |
| Detailed Procedure Used | |
| DP Revision Number | |
| Explanation of Sample Coding | |
| ***** See Attachment 1 for Information on Standards | |
| **Verification of ICP-AES Spectrometer** | |
| YMP ICP/AES ID# | |
| Notebook Procedure Used | |
| DP Revision Number | |
| ***** See Attachment 2 for Information on Standards | |
| **Vendor Sample Analysis Information** | |
| Vendor Analysis Notebook Reference | |
| Acceptance Plan Notebook Reference | |
| **Location of Data** | |
| Experimental Data (Notebook ID#) | |
| Experimental Data (Page(s) #) | |
| Reduced Data (Optical disk #) | |
| Reduced Data (Subdirectory) | |
| Reduced Data (File name) | |